MATERIALS PROPERTIES RESEARCH AT MSFC

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CTE Testing Requirements as Derived from SBMD/NMSD/AMSD Testing/Modeling

NGST

Motivation

- Two unexpected cryo deformations observed in SBMD:
  - *110 nm PV "dimple" near center.*
  - *134 nm PV, 16 nm RMS print-thru of rib structure.*
- Investigation led to large-scale CTE variation(s) as a "prime suspect" (nominal 293-30 K avg. CTE = 5.13 ppm/K):
  - *Can produce dimple with nonlinear, radial CTE variation of 0.6%.*
  - *Can produce print-thru with several CTE variations (X vs. Y, face vs. core, thin layer at face vs. bulk) with magnitudes ranging from 0.5% up to several %.*
- Brush CTE data (0-60 deg C) indicated possible XYZ variations of 0.8% with estimated measurement accuracy of +/-0.4%.
- Modeling of other systems also indicates %-level CTE variations can produce measurable impacts on surface figure.
- *Conclusion: Need more CTE data on ALL materials.*

Desired Measurement Accuracy

- Based on above, desire CTE data as function of position & direction to +/-0.05% (2.6 ppb/K & 0.7 ppm) for 293-30 K.

CTE Testing Approach

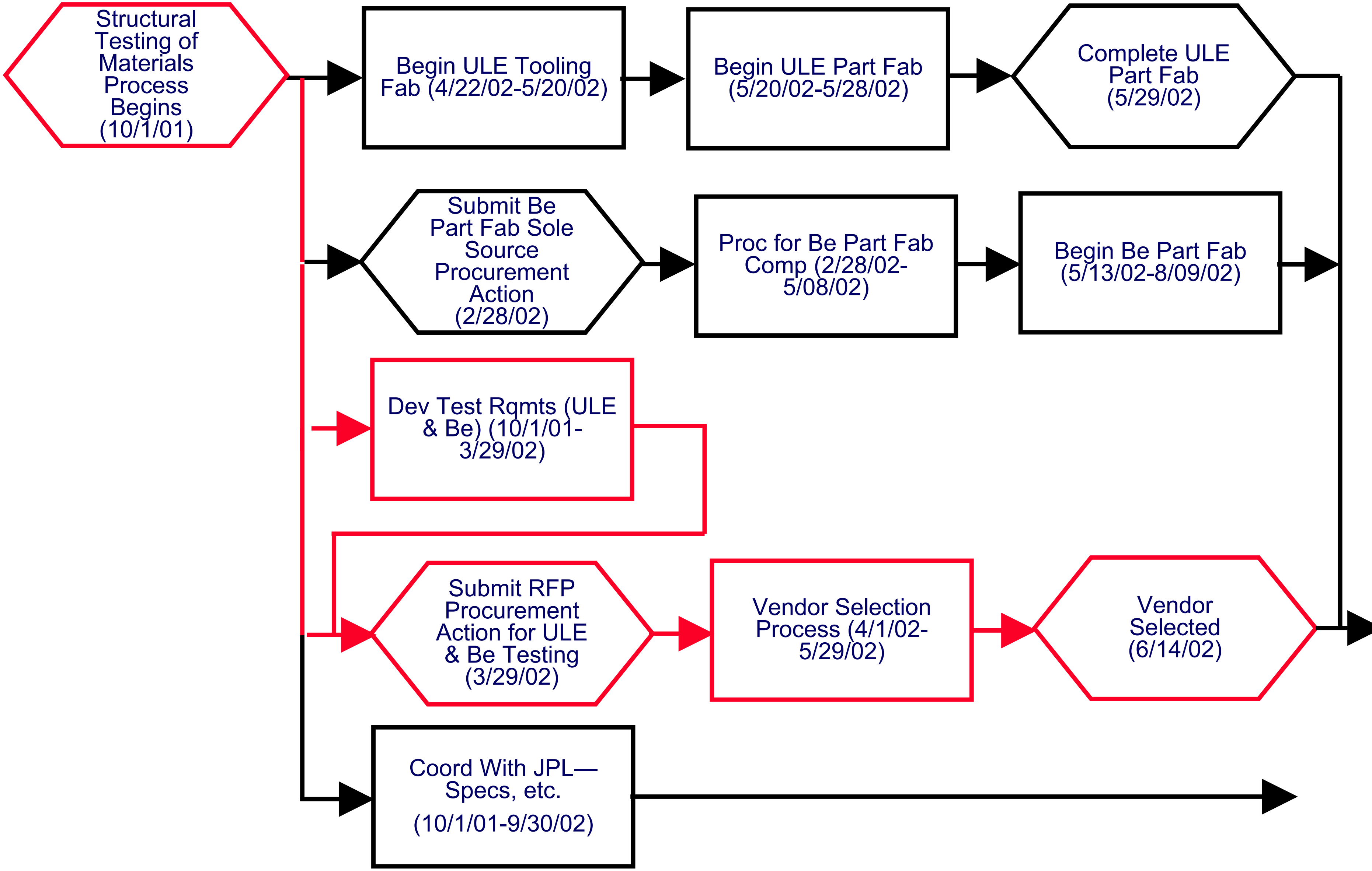
- Investigate currently available sources for ambient to 30K CTE measurements for O-30H Be & ULE.
- Talk directly with sources about their experience, methods, error sources, quality control, etc.
- Based on above, develop a minimum set of requirements for "first-round" CTE measurements in order to get *some* data into hands of AMSD modelers prior to cryo testing.
- Establish a weighted set of evaluation criteria in RFP to first-round vendors to encourage performance beyond minimum requirements.
- Continue searching for & evaluating sources (government & commercial) for higher-precision measurements.
- Select sources for "second-round" set of measurements to follow first round or when such sources become available.

Material Properties of Interest

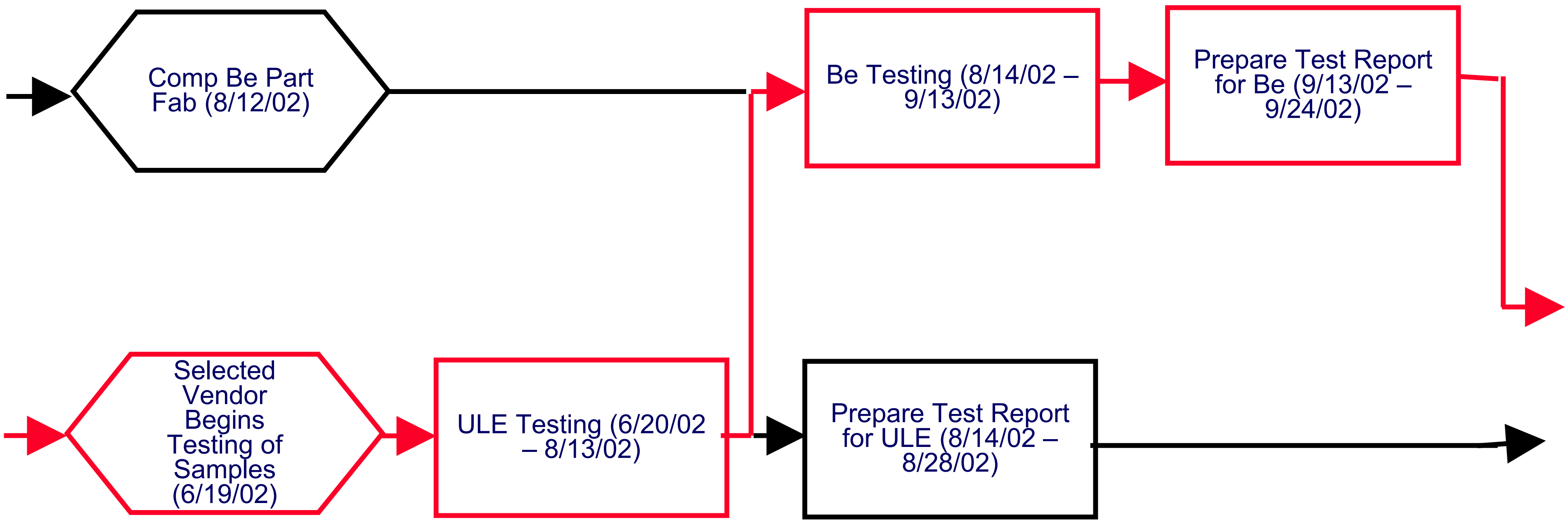
Materials Properties of interest for cryogenic applications

- **Primary**
  - *Coefficient of Thermal Expansion (CTE)*
  - *Uniformity of CTE*
- **Secondary**
  - *Thermal Conductivity*
  - *Specific Heat*
  - *Surface Emissivity & Absorptivity*
  - *Dynamic Dampening Coefficient*
  - *Tensile Strength*
  - *Stiffness & Creep*

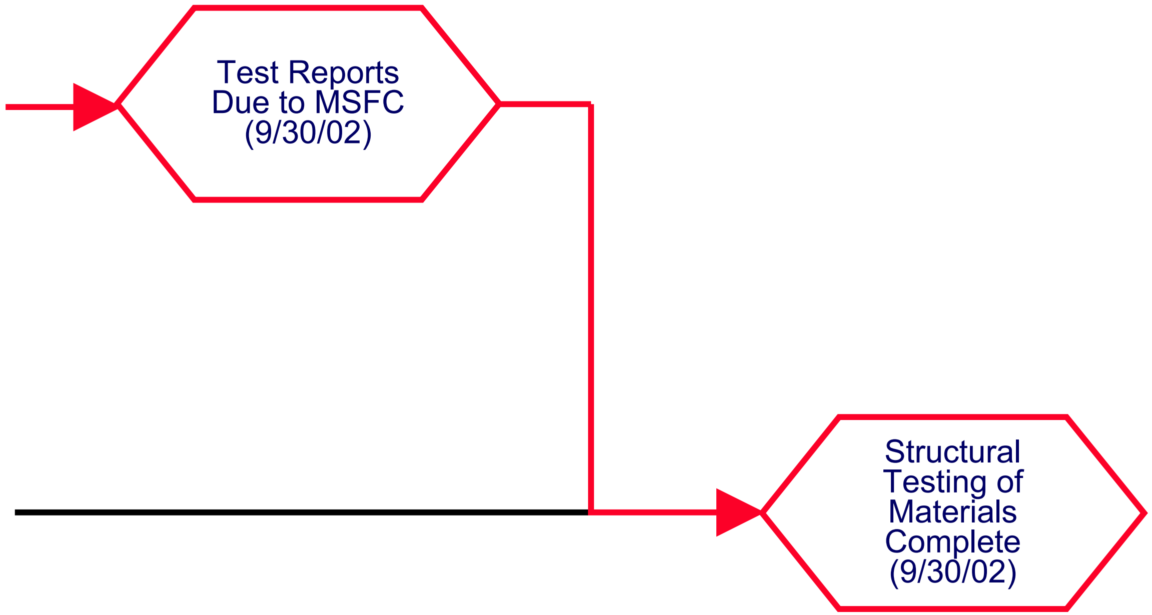
Overview of Activities for the Remainder of the Fiscal Year



Overview of Activities for the Remainder of the Fiscal Year, contd.



Overview of Activities for the Remainder of the Fiscal Year, contd.



Sources for CTE Measurements

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Sources for CTE Measurement								
Vendor	**Point of contact**	**Phone/e-mail**	**CTE Technique**	**Accuracy**	**Sample config.**	**Temp. range**	**ROM cost**	**Remarks**
Composit Optics, Inc. (COI)	Gregg McNab	858-621-6910 gmcnab@coi-world.com	Laser optical reflection from tilting mirror resting on samples and Zerodur reference.	+/- 1.8 microstrain	Two per reference; two sets per run	Room temp to 13 K	\$2500 per run (\$625 per sample)	COI performed CTE testing of MSFC beryllium in 1998
Southern Research Institute (SRI)	Jim Tucker	205-581-2526 tucker@sri.org	Laser interferometry	+/- 2 microstrain	One sample per run	Room temp to 30 K	\$2000 per run (\$2000 per sample)	MSFC Materials Dept. has standing work order with SRI, easy procurement
Precision Measurements and Instruments Corp. (PMIC)	Eileen Nicholson, Darrell Oakes	541-753-0607 info@pmiclab.com	Michelson laser interferometry	+/- 1 to 2 microstrain	Two samples per run	Room temp to 4 K	\$2200 per run (\$1100 per sample)	
Advanced Materials Lab, Inc. (AML)	Thomas Altshuler	978-369-9033 aml@tiac.net	Push rod measuring capacitance changes	+/- 7 microstrain	One sample per run	Room temp to 10 K	Unknown	

Note: These are only ROM costs. Some vendors have said they may be able to reduce costs depending on test requirements, temperatures, number of samples, system improvements, etc.

First-Round CTE Testing Minimum Requirements

Temperature Measurements

- Test between room temp (nominally 293 K) & a min temp no warmer than 30 K
- Min of two temp sensors/sample - must provide calibration data & evidence of no adverse effects on samples
- Measure sample temps with accuracy of +/-0.5 K or better
- Temp gradients in sample (during measurements) <=1.0 K

Distortion Measurements

- NOTE: “Distortion” defined in RFP as a change in sample length (either expansion or contraction) with temp
- Measure distortion of 1.5” Be & 1.0” ULE samples with accuracy of +/-5 micro-inch or better (*or +/-3 & +/-5 ppm*)
- State achievable accuracy for each sample type over above temp range & whether it is dependent on sample length or temp (if so, quantify vs. length/temp)

First-Round CTE Testing Minimum Requirements

Data Reporting (each sample)

- Room temp length before & after each cryo cycle
- Temp/distortion during either cool-down, warm-up, or both
  - *at least every 5 K between min temp & 100 K*
  - *at least every 10 K between 100 K & room temp*

Other Supporting Data

- Tests on calibrated samples (NIST, etc.)
- Tolerances & error source analyses for equipment/procedures
- Test first sample or sample set twice (remove & replace) to show repeatability of equipment/procedures

First-Round CTE Testing Deliverables

- Temperature sensor calibration
- Temperature sensor accuracy
- Analysis of temp sensor mounting effects on samples
- Room temp length measurement accuracy
- Distortion measurement accuracy for each sample type, vs. temp &/or vs. sample length, if variable
- Pre- & post-cryo cycle RT length for each sample
- Distortion vs. temperature data for each sample (& any reference samples used)
- Available calibration test data using NIST standard(s)
- Available tolerance & error source analyses
- Final report (including description of method/set-up, procedures, analyses, & data/results)
- Return of all samples

First-Round CTE Testing Evaluation Criteria

Beryllium CTE Testing Evaluation Criteria	Points
1) Temperature Measurement	
Low temperature no warmer than 30 K (colder is better)	2
Provide calibration data for temperature sensors	5
Minimum two temperature sensors per sample (more is better if there is no effect on sample behavior)	5
Analysis/evidence that temperature sensors do not affect thermal distortion behavior of samples	7
No temperature gradients in samples greater than 1.0 K (smaller is better)	8
Measure sample temperature with accuracy of +/- 0.5 K or better (smaller is better)	8
2) Room Temperature Length Measurement	
Measure sample at room temperature with accuracy of +/- 0.0001 inch or better (smaller is better)	2
3) Distortion Measurement	
Measure distortion with accuracy goal of +/- 5×10^{-6} inch or better (smaller is better)	30
Quantify distortion measurement accuracy versus sample length variation	8
Quantify distortion measurement accuracy versus temperature variation	8
4) Data Reporting	
Record temperature/distortion data both warm-to-cold and cold-to-warm (optional)	4
Record temp/distortion at least every 5 K for coldest to 100 K, at least every 10 K for 100 K to room temp (more often is better)	3
5) Other	
Test data on calibrated CTE standards	5
Tolerances/error contributions of test apparatus and procedures	5
Total	100

Backup Information

Questions Asked of First-Round CTE Measurement Vendors

How is temperature maintained? Do you use expendable gases or non-expendable cooling system (helium refrigerator)? Do you control the temperature during cool-down or during heat-up or both?

To what accuracy is temperature maintained (ex: +/- 0.5 K)? Are your silicon diodes calibrated against a known standard (they are known to have some variation from diode to diode)? How many silicon diodes do you normally use? How many can you fit onto our samples?

When taking data on a cryo run do you stop at specific temperatures to take data (and for how long normally) or just take it as the sample passes through that temperature? How do you know there are no temperature gradients in the sample when you take the data?

At what rate do you cool down and heat up? How long does it take?

How many data points do you typically take from room temp to cryo?

Questions Asked of First-Round CTE Measurement Vendors

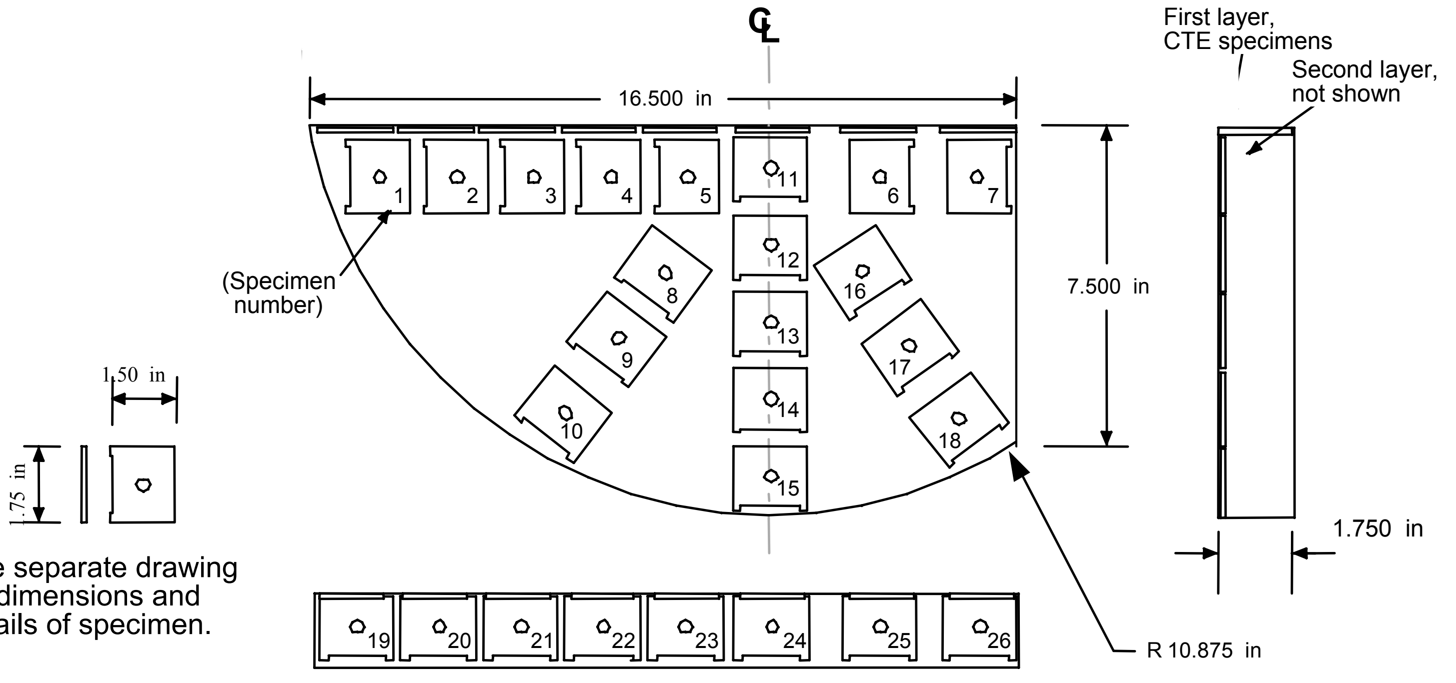
You state that you can measure CTE to X ppb/K. This is calculated from the slope of the microstrain vs. temperature data. With what accuracy, in microstrain, can you measure the thermal contraction/expansion of the sample? Can you give a specific example, such as measured within +/-20 microinches for a 6” sample from 293 K to 273 K?

Have you calibrated your system against a NIST CTE sample (SRM736 copper, for example) or some other standard? Do you have a listing of the different error sources in your system and their effects on the overall measurement error of the system?

Can you test samples as small as ours? How much extra cost are we looking at for our small samples? Explain how you test such small samples.

- *1” long x 1/2” wide x 1/8” thick ULE – may have low signal to noise ratio*
- *1 1/2” long x 1 3/4” wide x 1/8” thick beryllium – integrated CTE is about 5 ppm/K*

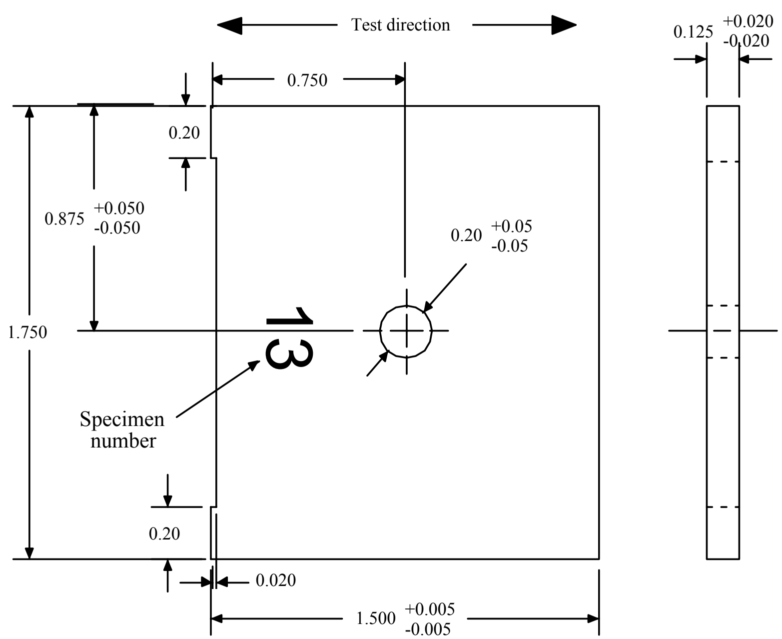
Be 0-30H Sample Layout



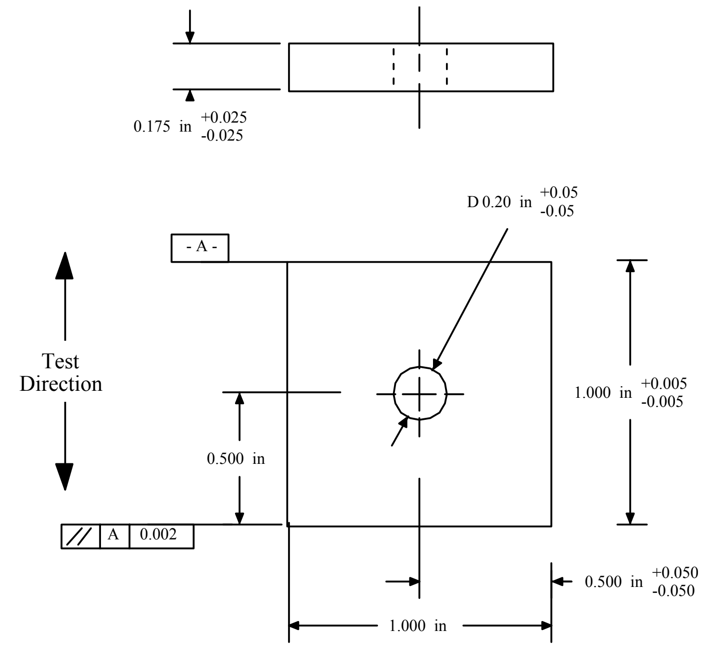
See separate drawing for dimensions and details of specimen.

The intent of this layout is to get as many samples as possible (in both the in-the plane direction and the through-the-thickness direction) radiating out from the center of the original billet. Supplier may adjust layout as necessary to fit all samples. Specimen numbers shown on this diagram shall be marked on both sides of each specimen using permanent ink (not etched) as shown in the specimen detail drawing. The purpose of this labeling is to maintain traceability to original location and orientation of specimens in billet

First-Round CTE Test Samples



Beryllium Sample

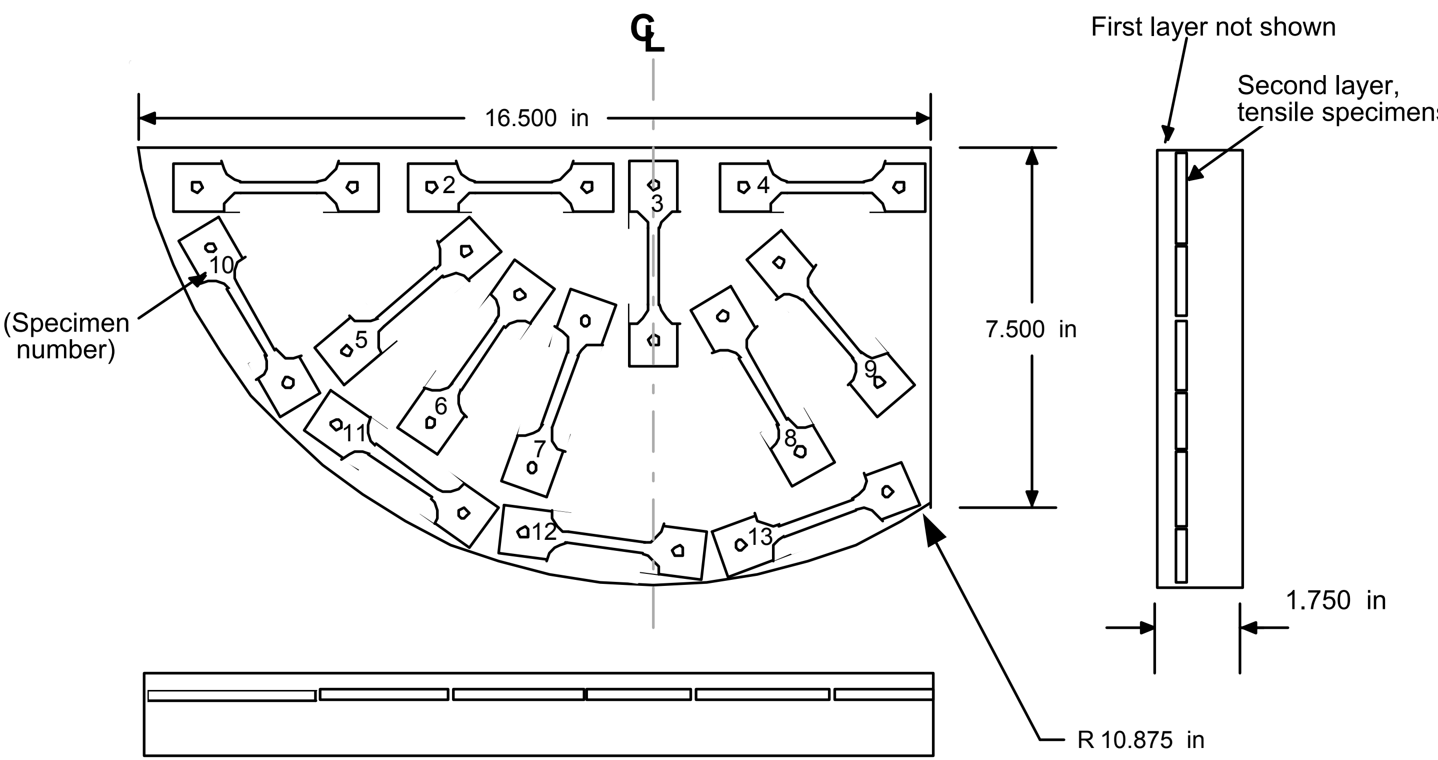


ULE Sample

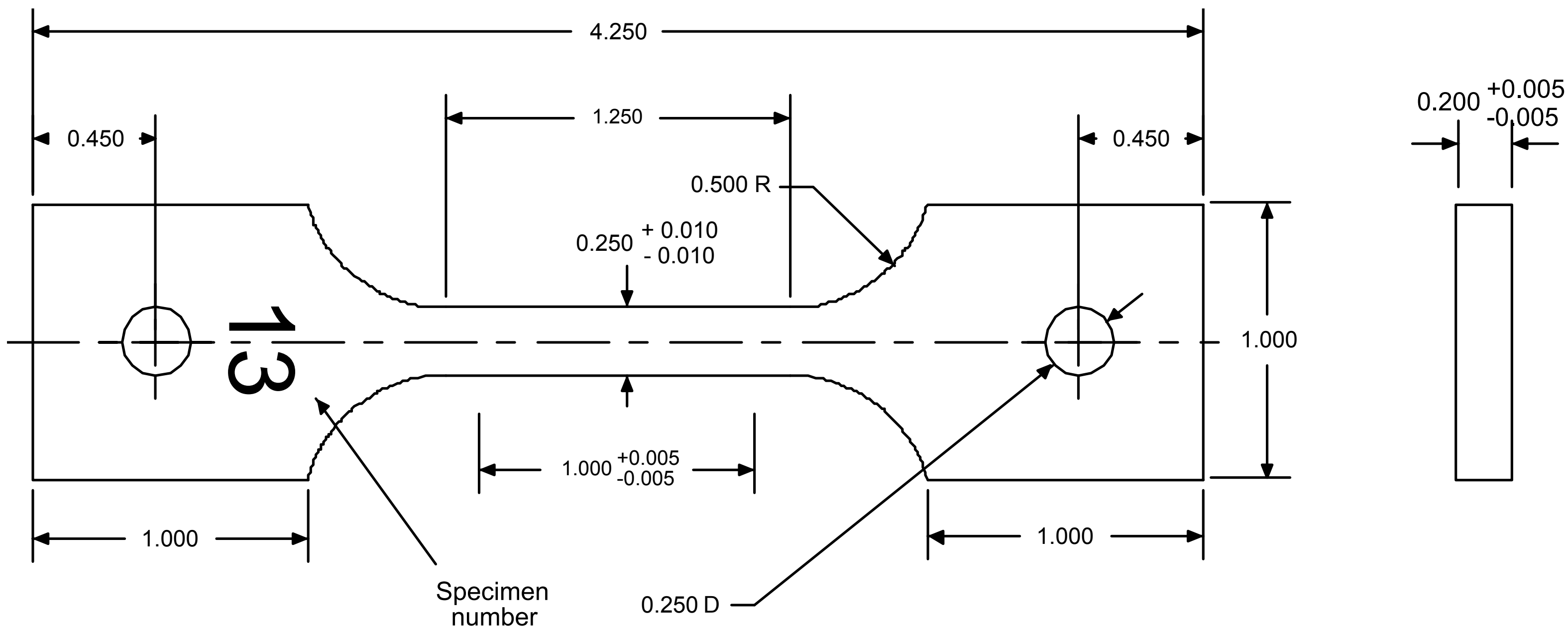
Be 0-30H Tensile Test Sample Layout

Tensile Test Specimen Layout (approximate) - Rev. A, 16 Jan 02

Second "layer" shown (CTE specimens taken from top layer, not shown)



Be 0-30H Tensile Test Specimen Detail

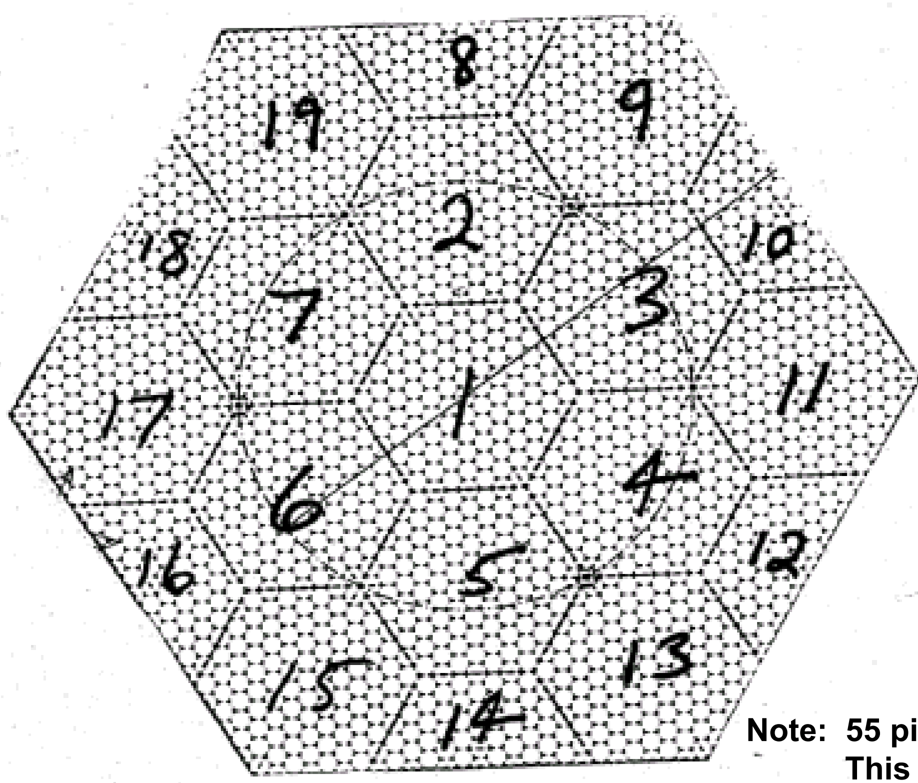


1. All dimensions are in inches.
2. Ends of the reduced section shall differ by not more than 0.002. There must be a gradual taper in width from the ends to the center (0.005 max).
3. Holes must be on center line of reduced section within +/- 0.002.
4. Deburr all edges.
5. Ends parallel within 0.001 inch.
6. Mark specimen number as shown on both faces of specimen using permanent ink.

7. Surface finish 63 overall; 32 in the 1.250 inch reduced section.

ULE Sample Detail

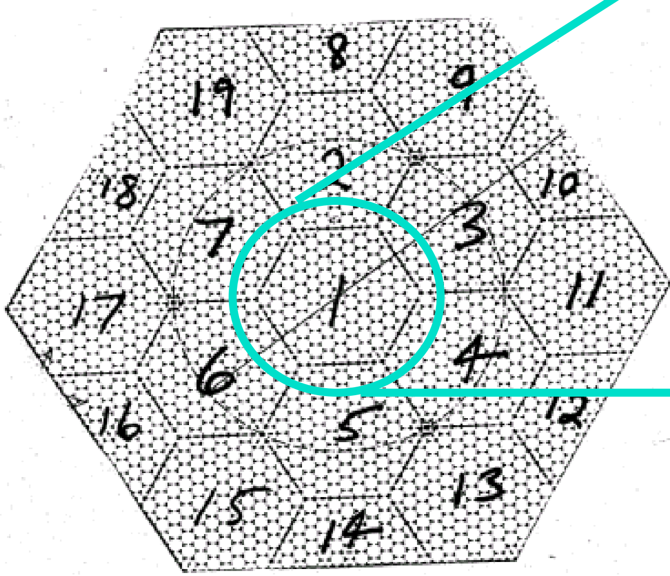
Overview of Kodak AMSD



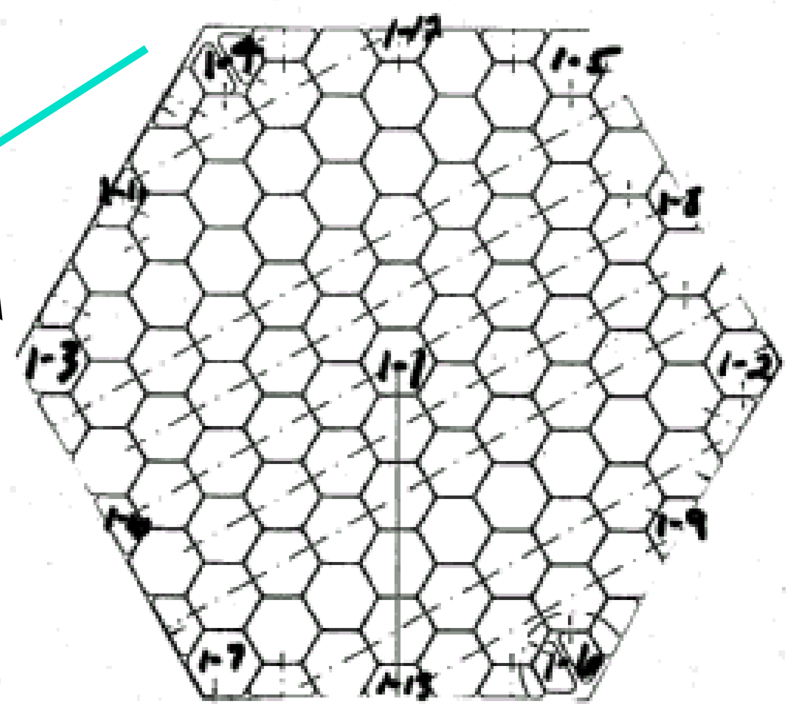
Note: 55 pieces total are selected for testing. This includes 19 segments or partial segments.

ULE Core Residual Locations

Note: CTE samples to be cut from Kodak AMSD core residuals at designated locations.



Segment 1



ULE Core Residual Locations